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古蔺雪胆中的新三萜皂苷*

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摘要：从采自四川汉源县的古蔺雪胆（*Hemsleya penxianensis* var. *gulinensis*）中分到 9 个三萜皂苷化合物，通过化学反应和光谱方法鉴定了它们的结构。其中 7 个为已知化合物，分别为齐墩果酸-28-O-β-D-吡喃葡萄糖苷（1），3-O-β-D-吡喃葡萄糖醛基齐墩果酸苷（3），3-O-β-D-吡喃葡萄糖醛基－齐墩果酸-28-O-α-L-吡喃阿拉伯糖苷（4），3-O-β-D-吡喃葡萄糖醛基－齐墩果酸-28-O-β-D-吡喃葡萄糖苷（5），3-O-α-L-阿拉伯糖基-(1→3)-β-D-吡喃葡萄糖醛基－齐墩果酸-28-O-β-D-吡喃葡萄糖苷（6），3-O-(6′-丁酯)-β-D-吡喃葡萄糖醛基－齐墩果酸-28-O-α-L-阿拉伯糖苷（7），3-O-(6′-丁酯)-β-D-吡喃葡萄糖醛基－齐墩果酸-28-O-β-D-吡喃葡萄糖苷（8）。两个新化合物，即雪胆皂苷 A（2）和雪胆皂苷 B（9）。

关键词：古蔺雪胆；葫芦科；齐墩果酸苷；雪胆皂苷 A 和 B
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New Triterpenoid Glycosides from *Hemsleya penxianensis* var. *gulinensis*

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Abstract：Two new triterpenoid saponins were isolated from the roots of *Hemsleya penxianensis* var. *gulinensis* together with seven known saponins oleanolic acid 28-O-β-D-glucopyranoside **(1)**，oleanolic acid 3-O-β-D-glucuropyranoside **(3)**，3-O-β-D-glucuropyranosyl-oleanolic acid-28-O-α-L-arabinopyranoside **(4)**，3-O-β-D-glucuropyranosyl-oleanolic acid-28-O-β-D-glucuropyranoside **(5)**，3-O-α-L-arabinopyranosyl-(1→3)-β-D-glucuropyranosyl-oleanolic acid-28-O-β-D-glucopyranoside **(6)**，3-O-(6′-butyl ester)-β-D-glucuropyran-osyl-oleanolic acid-28-O-α-L-arabinopyranoside **(7)**，3-O-(6′-butyl ester)-β-D-glucuropyranosyl-oleanolic acid-28-O-β-D-glucopyranoside **(8)**. The structures of determined as：oleanolic acid 28-O-β-D-glucopyransyl-(1→6)-β-D-glucopyranoside **(2)**，3-O-α-L-arabonipyranosyl-(1→3)-(6′-butyl ester)-β-D-glucuropyranosyl-oleanolic acid-28-O-β-D-glucuropyranoside **(9)**. Their structures were determined on the bases of the spectral and chemical evidences.

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Key words: *Hemsleya penxianensis* var. *gulinensis*; Cucurbitaceae; Hemslonin A and Hemslonin B

Hemsleya penxianensis var. *gulinensis* widely distributed in Sichuan province grows in the subtropical region at altitudes about 1500 – 2000 meters. This plant is used as herb medicines, for treatment of bronchitis, bacillary dysentery, and tuberculosis. Study on this plant led to the isolation of two new compounds, Hemslonin A, B together with seven known compounds, oleanolic acid 28-O-β-D-glucopyranoside (**1**), oleanolic acid 3-O-β-D-glucuropyranoside (**3**), 3-O-α-L-arabinopyranosyl-(1→3)-β-D-glucuropyranosyl-oleanolic acid-28-O-β-D-glucopyranoside (**6**) (Nie *et al*, 1984), 3-O-β-D-glucuropyranosyl-oleanolic acid-28-O-α-L-arabinopyranoside (**4**), 3-O-β-D-glucuropyranosyl-oleanolic acid-28-O-β-D-glucuropyranoside (**5**) (Shi *et al*, 1995), 3-O-(6′-butyl ester)-β-D-glucuropyranosyl-oleanolic acid 28-O-α-L-arabinopyranoside (**7**), 3-O-(6′-butyl ester)-β-D-glucuropyranosyl-oleanolic acid 28-O-β-D-glucopyranoside (**8**) (Chen, 2001).

Hemslonin A (**2**), white crystals, had a molecular formula of $C_{42}H_{68}O_{13}$ deduced from negative HRFABMS (obsd 779.459834, calcd 779.458171). Its FAB-MS also exhibited the fragment ions at m/z 617 $[M-1-162]^-$ and 455 $[M-1-162-162]^-$ (100). The ^{13}C NMR spectrum showed two anomeric carbon signals (δ 95.80, 104.70) and the methine signal (δ 79.78, C – 3), suggesting that the glucose only linked with C – 3 position. The spectra is similar to those of 1 expect the signals of additional glucose, suggesting that compound 2 had the same skeleton and substitutied groups as compound 1. Alkaline hydrolysis of compound 2 yields only glucose. The ^1H NMR spectra showed two anomeric protons at δ_H 4.41 (d, 1H, J = 8.1 Hz) and δ_H 5.41 (d, 1H, J = 7.8 Hz), indicated that two glucopyranosyl groups are in β configuration. The 2D NMR spectra data suggested that one glucopyranosyl (C – 6) is linked with the (C – 1) glucopyranosyl. Thus, compound 2 was determined as oleanolic acid 28-O-β-D-glucopyransyl-(1→6)-β-D-glucopyranoside.

Hemslonin B (**9**), was a white powder. Its molecular formula was determined as $C_{51}H_{82}O_{18}$ by HRFAB-MS (neg.) at m/z 981.4269 (calcd 981.5423). The FAB-MS also displayed $[M-1-162]^-$ (100), $[M-1-162-132]^-$ and $[M-1-162-132-232]^-$. The spectra is similar to those of **8** expect for the signals at δc 66.18 (CH_2), 31.7 (CH_2), 18.94 (CH_2), 14.09 (CH_3) in the ^{13}C NMR spectrum, suggesting that compound **9** had the same skeleton and substitutied groups as compound **8**. The ^1H NMR spectra showed the signals at δ_H 6.28 (d, 1H, J = 7.3 Hz), 5.31 (d, 1H, J = 7 Hz) and 4.94 (d, 1H, J = 7.8 Hz), indicated that glucopyranosyl, glucuropyranosyl, and arabinopyranosyl are in the β, β, and α configurations, respectively. The 2D NMR also showed that the C – 6 of the glucuronic acid had formed ester and the α-L-arabinopyranosyl unite was attached to C – 3 of the glucuronic acid. Thus the structure of **9** was assigned as oleanolic acid 28-O-β-D-glucuropyranosyl-3-O-α-L-arabonipyranosyl-(1→3)-(6′-butyl ester)-β-D-glucuropyranoside.

The structures of compounds **1**, **3 – 8** were identified by comparing their physical and spectral data with those reported in literatures.

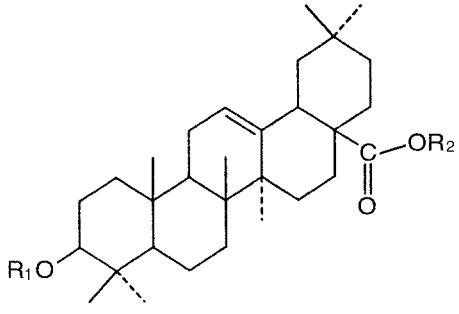
Experimental

General　All melting points were measured on an XRC－1 micro melting point apparatus and uncorrected. Optical rotation was taken on a SEPA－300 polarimeter. IR spectral data were measured on a Bio-Rad FTS－135 spectrometer with KBr pellets. MS spectra were recorded on a VG Auto Spec－3000 spectrometer NMR pectra were run on a Bruker AM－400 and a DRX－500 instrument with TMS as internal standard. CC were carried out with silica gel (Marine Chemical Industry Factory, Qingdao). The spots were visualized by spraying with 5% H_2SO_4 followed by heating.

Plant material　*Hemsleya penxianensis* var. *gulinensis*, Hanyuan, Sichuan, China. Specimen was taxonomically identified by Prof. Wen-Jin Zhang, Penxian Institute of Traditional Medicine, Sichuan.

Extraction and separation　The dried and powdered rhizomes (4 kg) were extracted with hot methnol for three times. The extract was evaporated to dryness in *vacuo*. A suspension of this extract (1.13 kg) in H_2O was extracted with *n*-BuOH saturated with H_2O. The *n*-BuOH layer was concentrated to dryness to give a crude saponin fraction (325 g).

The *n*-BuOH extract (325 g) was dissolved in water and was subjected to macroporous absorption resin D－101, eluting with EtOH-H_2O (0, 10%, 20%, 40%, 60%, 80%, 100%). These fractions were further purified by repeated column chromatography on Si gel and recrystallization to yield **1** (10 mg), **2** (30 mg), **3** (50 mg), **4** (16 mg), **5** (30 mg), **6** (40 mg), **7** (18 mg), **8** (20 mg), **9** (100 mg).



	R_1	R_2
1	H	—glc
2	H	—glc$^{6\rightarrow1}$glc
3	—glcUA	H
4	—glcUA	—ara
5	—glcUA	—glc
6	—glcUA$^{3\rightarrow1}$ara	—glc
7	6 glcUA (COOCH$_2$CH$_2$CH$_2$CH$_3$)	—ara
8	6 glcUA (COOCH$_2$CH$_2$CH$_2$CH$_3$)	—glc
9	6 glcUA$^{3\rightarrow1}$ara	—glc

ara: α-L-arabinopyranosyl
glc: β-D-glucopyranosyl
glcUA: β-D-glucuronic acid

Compound **1**, white needle crystals, $C_{36}H_{58}O_8$, MW = 618; mp 180－183℃ (decomposed); $[\alpha]_D^{26.1}$ + 21.74 (*c* 0.207, C_5D_5N); FAB-MS m/z (%): 617 $[M-1]^-$ (20), 455 $[M-1-162]^-$ (100); IR ν_{max}^{KBr} cm^{-1}: 3433, 2933, 2862, 1693, 1461, 1386, 1181, 1030, 996, 762; ^1H NMR (C_5D_5N, 400 MHz): δ 6.19 (1H, d, J = 8 Hz, glc H－1), 5.35 (1H, br, s, H－12), 1.18, 1.17, 1.13, 1.06, 0.91, 0.90, 0.86 (21H, s, 7 × CH_3); ^{13}C NMR data (C_5D_5N, 100.6 MHz) see Tables 1 and 2.

Compound **2**, white crystals, $C_{42}H_{68}O_{13}$, MW = 780; mp 245－250℃ (decomposed); $[\alpha]_D^{26}$ 18.52 (*c* 0.19, CH_3OH); FAB-MS m/z (%): 780 $[M]^-$ (38), 455 $[M-1-162-162]^-$ (100); HR-FAB-MS m/z (%) 779.4598 $[M-1]^-$; EI-MS m/z (%): 456 (7), 410 (5), 248 (100), 203 (84); IR ν_{max}^{KBr} cm^{-1}: 3644, 2926, 2836, 1725, 1638, 1464, 1390, 1197, 1071, 902; ^1H NMR (CD_3OD, 500 MHz): δ 5.41 (1H, d, J = 7.8

Hz, glc′ H－1), 5.32 (1H, d, H－12), 4.41 (1H, d, J＝8.7 Hz, glc-1), 4.18 (1H, d, J＝11.6 Hz, glc H－6), 3.91 (1H, d, J＝13.6 Hz, glc′ H－6), 3.83 (1H, dd, J＝6, 5.5 Hz, glc H－6), 3.73 (1H, dd, J＝5.5, 5.5 Hz, glc′ H－6), 1.22, 1.03, 1.01, 1.00, 0.97, 0.87, 0.84 (21H, s, 7×CH_3); ^{13}C NMR (CD_3OD, 125.8 MHz) see Tables 1 and 2.

Table 1 ^{13}C NMR data for the aglycone moieties of **1－9**

Position	1	2	3	4	5	6	7	8	9
1 (t)	39.49	39.87	39.40	39.47	38.81	38.87	39.28	39.25	40.17
2 (t)	28.77	28.87	28.99	29.01	30.96	28.39	28.70	28.78	26.70
3 (d)	79.41	79.78	89.83	89.86	89.14	89.40	89.68	89.64	91.28
4 (s)	39.87	40.67	40.18	40.32	39.61	39.67	40.05	40.05	40.84
5 (d)	56.33	56.84	56.57	56.55	55.93	55.97	56.32	56.29	57.15
6 (t)	19.33	17.87	19.22	19.28	18.83	18.73	19.03	19.03	17.75
7 (t)	30.47	33.52	33.95	33.97	33.37	33.40	33.75	33.72	33.54
8 (s)	40.44	40.64	40.18	40.66	40.05	40.13	40.42	40.44	40.83
9 (d)	48.66	47.90	48.72	48.78	48.13	48.31	48.56	48.57	47.98
10 (s)	37.87	38.22	37.65	37.73	37.07	37.13	37.52	37.50	37.89
11 (t)	24.15	24.48	24.58	24.56	23.86	23.90	24.34	24.32	24.02
12 (d)	123.41	123.83	123.24	123.74	123.13	123.12	123.45	123.38	123.72
13 (s)	144.66	144.93	145.59	144.98	144.27	144.40	144.76	144.71	144.88
14 (s)	42.87	42.79	42.73	42.48	41.90	41.98	42.24	42.27	42.60
15 (t)	28.59	28.86	28.99	27.41	26.51	26.56	27.17	27.17	28.83
16 (t)	23.92	24.48	24.58	24.02	23.56	23.63	23.77	23.93	24.02
17 (s)	47.51	47.20	47.34	47.97	47.17	47.35	47.72	47.53	47.18
18 (d)	42.27	42.60	42.88	42.90	42.28	42.36	42.66	42.66	42.80
19 (t)	46.74	47.10	47.48	47.00	46.42	46.49	46.77	46.74	47.18
20 (s)	31.26	31.62	31.71	31.65	30.96	31.00	31.42	31.41	31.54
21 (t)	34.50	34.92	35.00	34.88	34.17	34.23	34.64	34.54	34.50
22 (t)	33.03	33.92	33.95	33.53	32.69	32.76	33.27	33.06	33.14
23 (q)	29.26	28.78	28.99	29.00	28.43	28.39	28.79	28.68	28.58
24 (q)	17.02	16.35	17.74	17.77	17.20	17.22	17.47	17.46	17.01
25 (q)	16.13	16.08	16.21	16.31	15.73	15.77	16.09	16.10	16.06
26 (q)	18.02	17.87	18.17	18.24	17.63	17.69	17.98	18.00	17.75
27 (q)	26.59	26.32	26.97	26.91	26.33	26.37	26.66	26.67	26.47
28 (s)	176.94	178.14	181.43	177.40	177.38	175.27	177.12	177.00	177.98
29 (q)	33.61	33.52	33.95	33.97	33.37	33.40	33.75	33.72	33.54
30 (q)	24.33	24.08	24.58	24.47	23.86	23.90	24.24	24.21	24.03

(**1**, **3**, **4**, **5**, **6**, **7**, **8** in C_5D_5N and **2**, **9** in CD_3OD)

Compound **3**, white powders, $C_{36}H_{56}O_9$, MW＝632; $[\alpha]_D^{25.3}$ ＋8.35 (*c* 0.479, C_5D_5N); FAB-MS m/z (%): 631 (100) $[M-1]^-$ (100), 455 $[M-1-176]^-$ (10); EI-MS m/z (%): 456 (5), 412 (18), 248 (100), 203 (64), 163 (81); IR ν_{max}^{KBr} cm^{-1}: 3644, 2926, 2836, 1725, 1638, 1464, 1390, 1197, 1071, 902; ^{1}H NMR (C_5D_5N, 500 MHz): δ 5.65 (1H, br, s, H－12), 4.82 (1H, d, J＝7 Hz, glcUA H－1), 1.33, 1.32, 1.29, 1.04, 0.99, 0.98, 0.79 (21H, s, 7×CH_3); ^{13}C NMR (C_5D_5N, 125.8 MHz) see Tables 1 and 2.

Compound **4**, white needle crystals, $C_{41}H_{64}O_{13}$, MW＝764; mp 215－217℃ (decomposed); $[\alpha]_I^{25.4}$ 6.60 (*c* 0.482, CH_3OH); FAB-MS m/z (%): 763 (100) $[M-1]^-$ (100), 631 $[M-1-132]^-$ (10), 455 $[M-1-132-176]^-$ (7); IR ν_{max}^{KBr} cm^{-1}: 3434, 2940, 2836, 1741, 1595, 1463, 1388, 1160, 1075, 776; ^{1}H NMR (C_5D_5N, 400 MHz): δ 6.27 (1H, s, ara H－1), 5.40 (1H, s, H－12), 5.01 (1H, s, glcUA H－1), 1.29, 1.25, 1.01, 0.98, 0.94, 0.90, 0.80 (21H, s, 7×CH_3); ^{13}C NMR (C_5D_5N, 100.6 MHz) see Tables 1 and 2.

Compound **5**, white needle crystals, $C_{42}H_{66}O_{14}$, MW＝794; mp 235－238℃ (decomposed); $[\alpha]_D^{26.4}$ 14.60 (*c* 0.822, C_5D_5N); FAB-MS m/z (%): 794 $[M]^-$ (100), 631 $[M-1-162]^-$ (37), 455 $[M-1-162-176]^-$ (10); EI-MS m/z (%): 456 (2), 410 (8), 392 (11), 248 (100), 203 (82), 163 (25); IR ν_{max}^{KBr}

cm^{-1}：3415，2938，2848，1699，1606，1429，1386，1160，1024，947；^1H NMR（C_5D_5N，500 MHz）：δ 6.28（1H，d，J = 7.3 Hz，glc H－1），5.56（1H，s，H－12），4.44（1H，d，J = 11.1 Hz，glcUA H－1），1.28，1.25，1.07，0.95，0.93，0.89，0.79（21H，s，7 × CH_3）；^{13}C NMR（C_5D_5N，125.8 MHz）see Tables 1 and 2.

Table 2　^{13}C NMR data for the sugar moieties of **1－9**

Position		**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**
3-O-glcUA	1			107.32	108.07	106.73	106.35	107.85	107.88	105.58
	2			75.91	76.31	76.18	74.72	75.90	75.88	74.07
	3			78.76	78.55	75.35	84.30	78.50	78.49	86.38
	4			74.38	72.11	73.88	72.58	73.59	73.59	72.86
	5			76.99	78.95	78.18	76.30	77.83	77.83	76.30
	6			177.74	173.90	176.70	174.98	170.86	170.88	170.56
$COOCH_2CH_2CH_2CH_3$	α							65.49	65.48	66.18
	β							30.53	30.54	31.70
	γ							19.80	19.79	18.94
	δ							14.32	14.32	14.09
ara（1→3）	1						105.45			106.78
	2						71.92			71.48
	3						74.48			74.61
	4						69.42			69.65
	5						67.20			67.34
28-O-ara	1				96.63			96.35		
	2				68.86			71.84		
	3				74.71			74.33		
	4				74.27			68.58		
	5				66.97			66.68		
28-O-glc	1	92.26	95.80			95.88	95.98		96.27	95.67
	2	74.64	74.00			74.20	74.30		74.62	73.86
	3	78.60	75.17			78.95	79.04		79.38	78.96
	4	71.63	71.03			71.23	71.34		71.60	71.10
	5	79.82	78.09			79.42	79.49		79.84	78.60
	6	62.72	69.53			62.34	62.44		62.71	62.53
glc′(1→6)	1		104.70							
	2		77.86							
	3		78.04							
	4		71.61							
	5		78.24							
	6		62.79							

Compound **6**，white powders，$C_{47}H_{74}O_{18}$，MW = 926；mp 270－273℃（decomposed）；$[\alpha]_D^{26.3}$ + 7.67（*c* 0.391，CH_3OH）；FAB-MS m/z（%）：925 $[M-1]^-$（100），793 $[M-1-132]^-$（10），763 $[M-1-162]^-$（35），631 $[M-1-162-132]^-$（3），455 $[M-1-162-132-176]^-$（5）；EI-MS m/z（%）：456（4），438（3），412（7），392（15），248（100），203（76），163（58）；IR ν_{max}^{KBr} cm^{-1}：3418，2938，2848，1725，1640，1456，1364，1157，1078，946；^1H NMR（C_5D_5N，400 MHz）：δ 6.29（1H，d，J = 8 Hz，glc H－1），5.57（1H，br，H－12），5.43（1H，br，J = 7.4 Hz，glcUA H－1），5.24（1H，d，J = 8 Hz，ara H－1），1.29，1.28，1.08，0.96，0.94，0.90，0.82（21H，s，7 × CH_3）；^{13}C NMR（C_5D_5N，100.6 MHz）see Tables 1 and 2.

Compound **7**，yellow powders，$C_{45}H_{72}O_{13}$，MW = 820；$[\alpha]_D^{25.5}$ + 14.74（*c* 0.407，C_5D_5N）；FAB-MS m/z（%）：819 $[M-1]^-$（5），763 $[M-1-56]^-$（7），687 $[M-1-132]^-$（100），455 $[M-1-132-176-56]^-$（9）；IR ν_{max}^{KBr} cm^{-1}：3449，2927，2868，1742，1463，1368，1162，1073，825，775，629；^1H NMR（C_5D_5N，400 MHz）：δ 6.25（1H，d，J = 5.5 Hz，ara H－1），5.42（1H，s，H－12），4.96（1H，d，J = 7.6 Hz，glcUA H－1），1.29，1.27，1.01，0.95，0.94，0.92，0.82（21H，s，7 × CH_3），0.76（3H，t）；^{13}C

NMR (C_5D_5N, 100.6 MHz) see Tables 1 and 2.

Compound **8**, yellow powders, $C_{46}H_{74}O_{14}$, MW = 850; $[\alpha]_D^{24.5}$ + 12.67 (*c* 1.105, C_5D_5N); FAB-MS m/z (%): 850 [M]$^-$ (4), 793 [M − 1 − 56]$^-$ (7), 687 [M − 1 − 162]$^-$ (100), 455 [M − 1 − 162 − 176 − 56]$^-$ (21); IR ν_{max}^{KBr} cm^{-1}: 3401, 2925, 2859, 1737, 1461, 1372, 1162, 1070, 627; ^1H NMR (C_5D_5N, 400 MHz): δ 6.28 (1H, d, J = 8.1 Hz, glc H − 1), 5.40 (1H, s, H − 12), 4.96 (1H, d, J = 7.8 Hz, glcUA H − 1), 1.26, 1.25, 1.06, 0.94, 0.90, 0.86, 0.82 (21H, s, 7 × CH_3), 0.75 (3H, t); ^{13}C NMR (C_5D_5N, 100.6 MHz) see Tables 1 and 2.

Compound **9**, white powders, $C_{51}H_{82}O_{18}$, MW = 982; mp 198 − 200℃ (decomposed); $[\alpha]_D^{26}$ + 16.62 (*c* 0.361, CH_3OH); FAB-MS m/z (%): 981 [M − 1]$^-$ (7), 925 [M − 1 − 56]$^-$ (10), 820 [M − 1 − 161]$^-$ (100), 687 [M − 1 − 161 − 133]$^-$ (13), 455 [M − 1 − 161 − 133 − 176 − 56]$^-$ (20); EI-MS m/z (%): 642 (17), 456 (10), 439 (40), 423 (13), 393 (18), 248 (100), 203 (87), 159 (45), 133 (53); IR ν_{max}^{KBr} cm^{-1}: 3443, 2942, 2856, 1740, 1462, 1372, 1160, 1075, 781, 635; ^1H NMR (C_5D_5N, 400 MHz): δ 6.28 (1H, d, J = 8 Hz, glc H − 1), 5.41 (1H, s, H − 12), 5.31 (1H, d, J = 7 Hz, glcUA H − 1), 4.55 (1H, d, J = 8 Hz), 4.94 (1H, d, J = 7.8Hz, ara H − 1), 3.77 (1H, d, J = 11.1 Hz), 1.26, 1.26, 1.07, 0.95, 0.90, 0.87, 0.81 (21H, s, 7 × CH_3), 0.77 (3H, t); ^{13}C NMR (CD_3OD, 125.8 MHz) see Tables 1 and 2.

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